CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Oil Solvent

Dodecane with purity of more than 99% and specific gravity of 0.875 from Aldrich Chemical Company was used for preparing the model oils in the cold finger experiments.

3.1.2 Paraffin Waxes

There were two waxes used in this work; Wax No. 1 from Aldrich Chemical Company with melting point of 54 – 56°C and Wax No. 2 from Mobil Company with melting point of 60°C. The carbon number distributions of these two waxes were obtained by a Varian 3800 high temperature gas chromatograph (HTGC) and are shown in Figures 3.1 and 3.2. High temperature high-resolution gas chromatography is commonly used to characterize paraffin deposits in petroleum production pipelines (Neto *et al.*, 1994). Carbon number distribution of Wax No. 1 had a range from C23 to C38 while Wax No. 2 had the distribution in a range of C21 to C36.

3.1.3 Model Oils

Two model oils were prepared for use in the cold finger experiments. Model Oil No. 1 was the mixture of 5.5 wt.% Wax No. 1 in dodecane. Model Oil No. 2 was the mixture of 2.4 wt.% Wax No. 2 in dodecane.

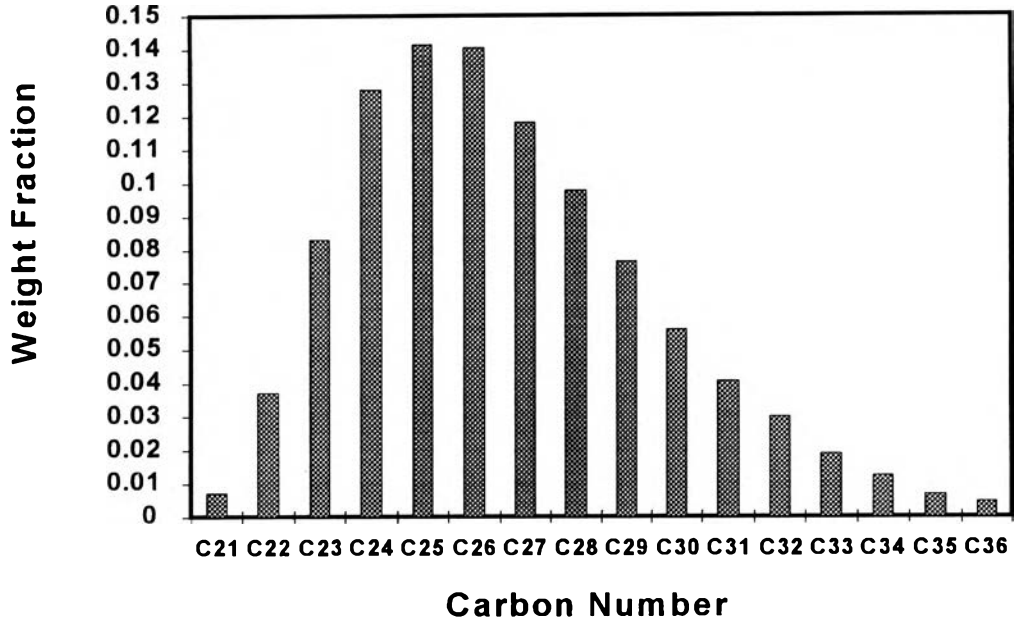


Figure 3.1 Carbon number distribution of Wax No. 1

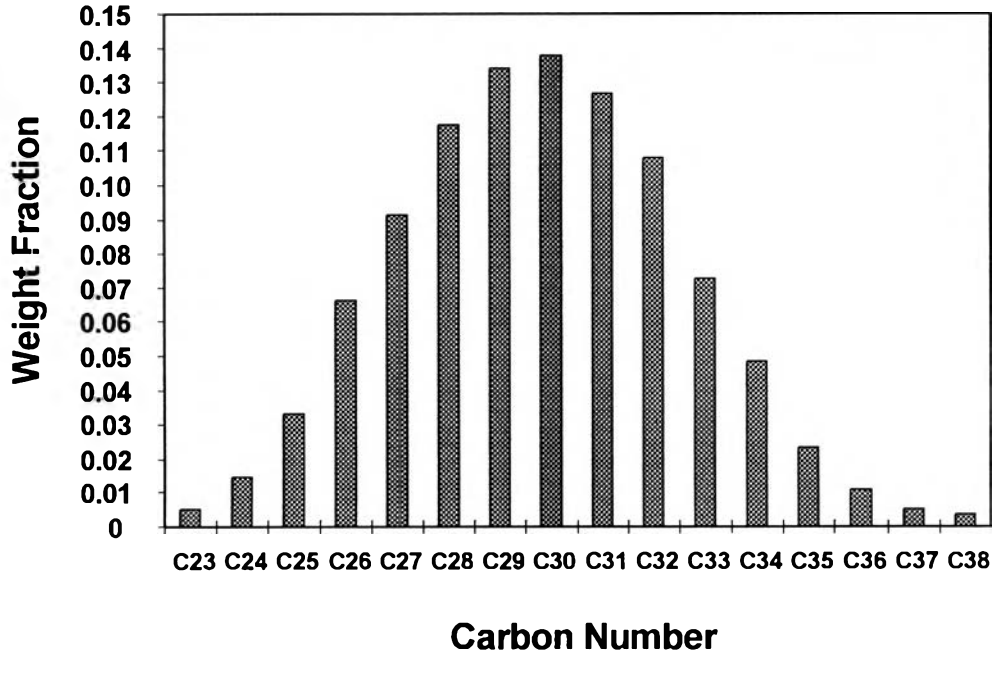


Figure 3.2 Carbon number distribution of Wax No. 2

3.2 Experimental Equipment

3.2.1 Cross-Polarized Light Microscope

The cross-polarized light microscope (Zeiss PhotoScope2) coupled with a temperature-control device (Figure 3.3) was used to measure cloud point of the synthetic wax-oil mixtures by detecting the formation of the first crystals. The temperature at which a wax-oil mixture becomes a saturated solution is called the cloud point temperature. When the temperature of the wax-oil mixture goes below the cloud point, waxes start precipitating out of the solution as wax crystals.

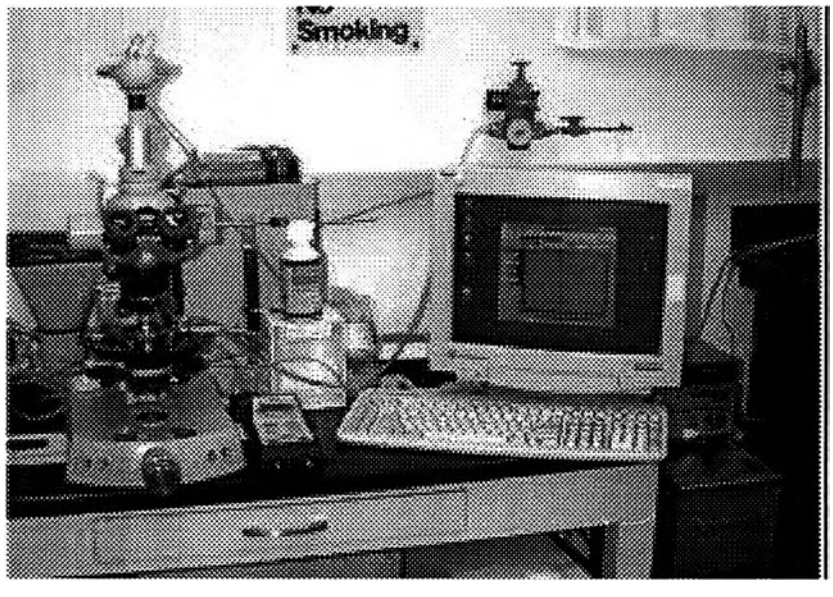


Figure 3.3 Cross-polarized light microscope apparatus

3.2.2 Cold Finger Setup

A laboratory cold finger setup was used to simulate the cold wall of the pipeline in the presence of warm waxy oil. Figure 3.4 shows a sketch of the cold finger apparatus, which was used to study the effect of temperature on the critical carbon number. It consisted of a temperature-

controlled cold steel cylindrical finger submerged in a bath of warm wax-oil mixture.

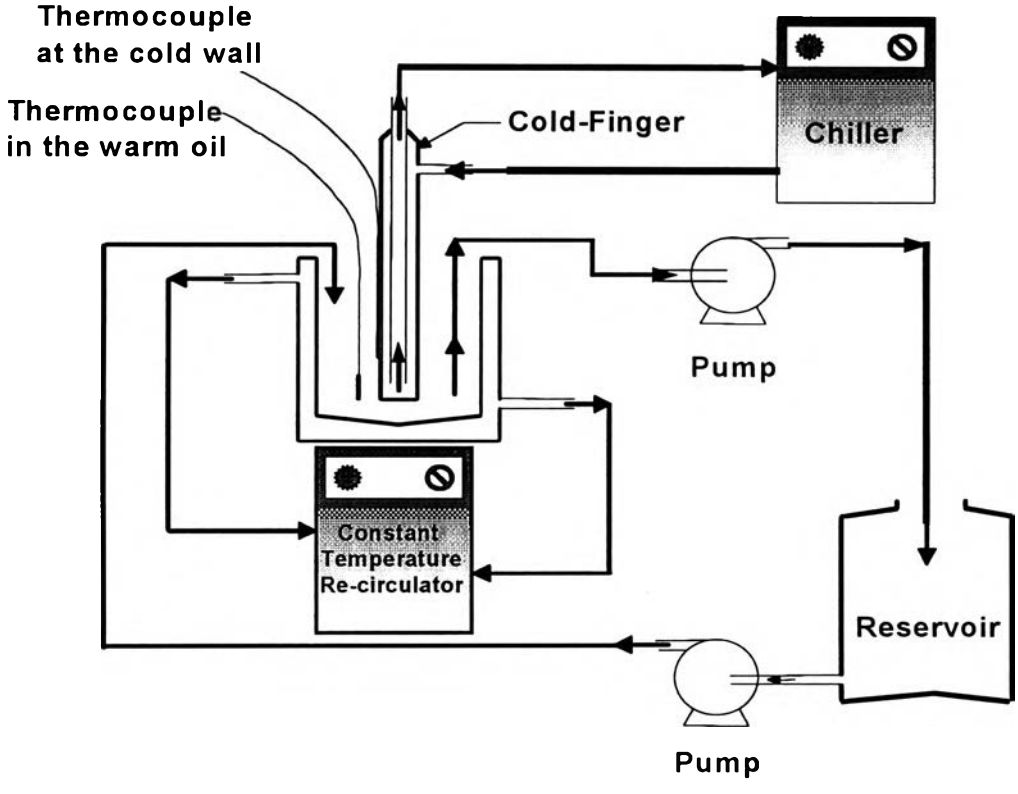


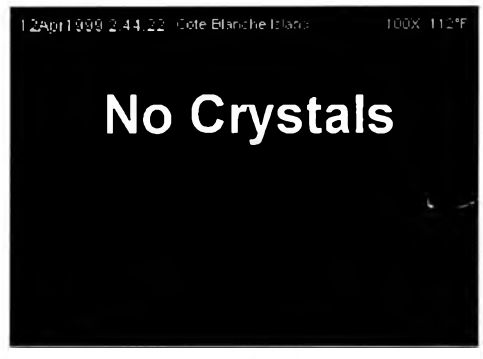
Figure 3.4 Cold finger apparatus

3.3 Expermental Procedure

3.3.1 Cloud Point Measurement

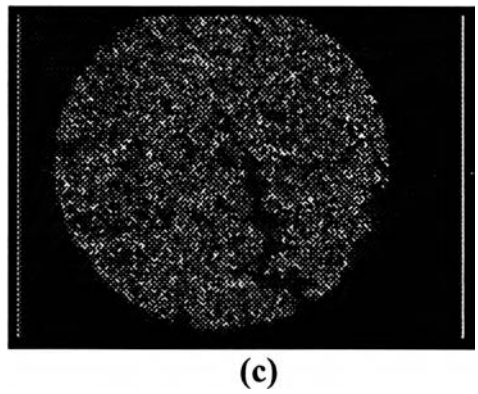
The wax-oil mixtures of various wax concentrations of either Wax No. 1 or Wax No. 2 in dodecane were prepared. Initially, wax-oil mixture was a clear solution at high temperatures. Then, the temperature was gradually decreased until the first wax crystals precipitated out, which could be observed by the cross-polarized light microscope (Figure 3.5). The

temperature at such condition was recorded as a cloud point temperature of the particular wax-oil mixture.



(a)

(b)



(c)

Figure 3.5 Cloud point measurement using cross-polarized light microscope: (a) $T > T_{cloud}$, (b) $T = T_{cloud}$, and (c) $T < T_{cloud}$

3.3.2 Cold Finger Experiment

A series of cold finger experiments were carried out using two model oils. Wax concentrations of both model oils were chosen to certain values corresponding to the cloud points of 20°C, which were obtained from the cloud point measurement. Water was circulated through the outer jacket of the bath using a constant temperature re-circulator to control the temperature of the wax-oil mixture at 25°C, i.e. 5°C above the wax-oil mixture cloud point. Chiller is used to maintain the temperature of the cold finger at 5°C, 10°C, and 15°C. The temperature of gel deposits (T_{gel}) is defined as the average temperature between the cold finger temperature (T_{wall}) and deposit-oil interfacial temperature (T_{int}), which is the cloud point of model oil, as shown in Figure 3.6. In order to keep the concentration of the wax in the warm wax-oil mixture constant throughout the experiment, a fresh wax-oil mixture was continuously circulated through the bath using two pumps (Milton Roy model CM4000). Three different experiments were conducted for 6 hours, 12 hours, and 24 hours. The gel deposit was collected from the cold finger after each experiment. Carbon number distribution of the gel deposit was analyzed by using High Temperature Gas Chromatography (HTGC) and the critical carbon number was then determined.

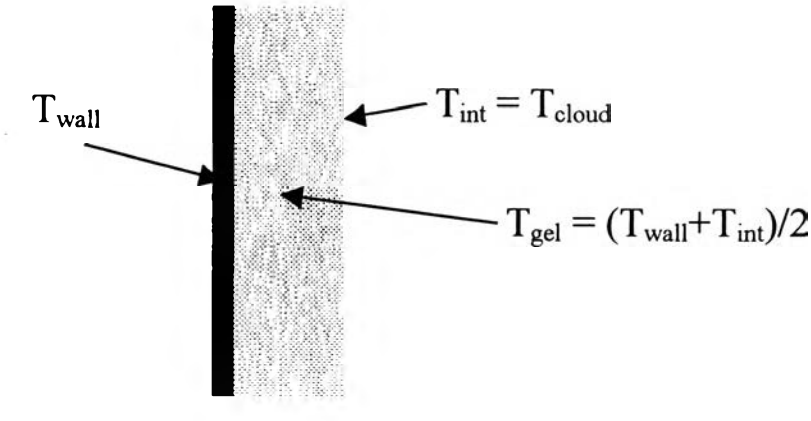


Figure 3.6 The temperature of gel deposit on wall of cold finger